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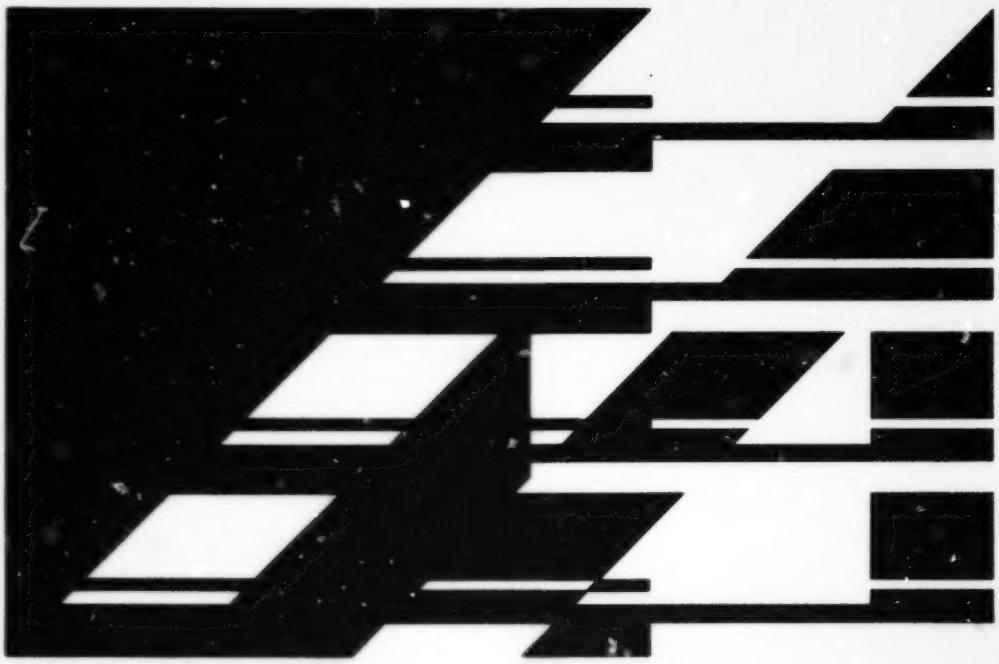

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SURFACE TENSION MEASUREMENT TECHNIQUES AS APPLIED TO FUSED SALT SYSTEMS AND A LITERATURE REVIEW OF SURFACE TENSIONS IN THE SYSTEMS $PbCl_2$-KCl-NaCl AND $PbCl_2$-KCl-LiCl

by

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SUMMARY

The techniques for surface tension measurements are reviewed, particularly those applicable to high temperature studies. Based on this review, a method is recommended for the surface tension measurements of the systems $PbCl_2$-KCl-NaCl and $PbCl_2$-KCl-LiCl. This is the maximum bubble technique.

The literature on the surface tension of the two systems has been reviewed. While no data exist for the ternary system, the data for the respective binaries are well established. Based on this knowledge, surface tensions in the ternary systems have been estimated.

A REVIEW OF THE TECHNIQUES FOR SURFACE TENSION MEASUREMENTS

INTRODUCTION

The purpose of this section is to review the existing methods for the measurements of surface tension and to determine which technique is most suitable for the application to molten salt systems.

The most common methods are: (1) capillary rise method; (2) maximum bubble-pressure method; (3) detachment methods; (4) drop weight method; and (5) profile measurement methods.

Capillary Rise Method

The basic observation is that, upon immersing an open vertical capillary tube into a liquid which wets the tube walls, the liquid will naturally rise to a maximum height, h (see Fig.1), from which the surface tension of the liquid, γ, can be calculated according to Equation (1).

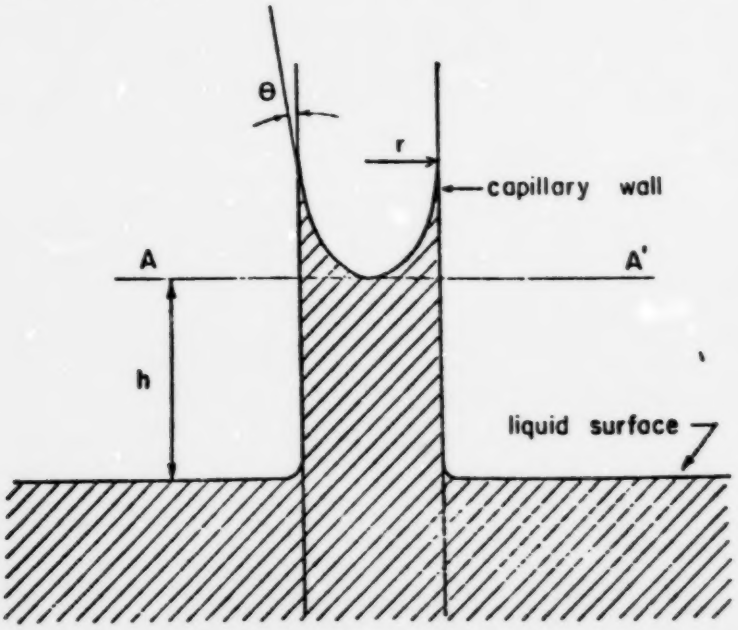

Fig.1. Capillary Rise Method

$$\gamma \cos \theta = \Delta\rho.g.h.r/2 \qquad (1)$$

where θ = contact angle, $\Delta\rho$ = the difference in densities between the liquid and gas phase, and r = capillary radius.

This equation is only an approximate solution, since it fails to include the volume of liquid above the line AA' of Fig.1. The exact treatment of the phenomenon must take into account the shape of the meniscus. The correction factors have been devised by Rayleigh(1) and Sugden(2) for the specific case when θ is zero.

This method is suited for systems where visual observation of the liquid is possible, and has been used mainly for relatively low temperature measurements.

The fatal weak point of the method is that it can only be used with certainty in systems where the liquid completely wets the capillary walls, i.e. $\theta = 0$. Otherwise, θ must be measured, and this is difficult to do accurately.

Maximum Bubble-Pressure Method

This method involves the very slow formation of a bubble at the tip of a capillary immersed in the melt and the subsequent determination of the maximum pressure in the bubble at the very instant it bursts. The relationship between the maximum pressure, P_{max}, and the surface tension, γ, is represented by the following equation:

$$P_{max} = P_H + P_\gamma = \Delta\rho.g.H + 2\gamma/r \qquad (2)$$

where $\Delta\rho$ = the difference in density between the liquid and gas, H = immersion depth of the capillary into the melt, and r = capillary radius (Fig.2).

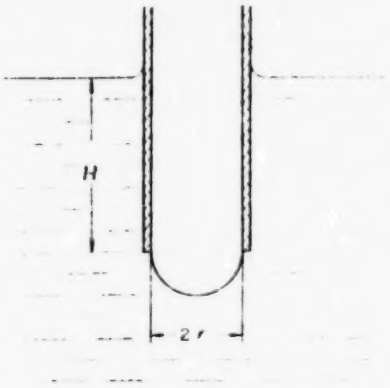

Fig.2. Maximum Bubble-Pressure Method

The equation applies only for very small bubbles where the bubble shape is hemispherical at its point of instability. For larger diameter tubes, the bubble assumes a deformed shape, due to the difference in hydrostatic head between the tip of the capillary and the bottom of the bubble.

Correction tables(3,4) and revised equations(5,6,7) have been proposed for relatively large diameter capillaries. Equation (3) was proposed by Schrödinger(7):

$$\gamma = \frac{r.P_\gamma}{2} \left\{1 - \frac{2}{3}\left(\frac{r.\rho.g}{P_\gamma}\right) - \frac{1}{6}\left(\frac{r.\rho.g}{P_\gamma}\right)^2\right\} \qquad (3)$$

where ρ is the density of liquid. This equation is valid in the region $r \leq 0.2\sqrt{2\gamma/\rho.g}$.

The liquid density, which is required for the calculation, can be obtained as a part of the same experiment from the dependency of P_{max} on H.

An important variation of this method is the twin capillary method investigated by Sugden(4), where two capillary tubes of different diameter were immersed to the same depth. It does not require knowledge of the liquid density, nor does it require the measurement of the capillary immersion depth. Thus, these sources of errors are virtually eliminated. However, the method does require measurement of two capillary radii and identical depth of immersion for each capillary tube, which are difficult to do and would be the major error sources.

There may be a cooling effect at the surface of the liquid during the formation and release of the bubbles from the capillary. For slow bubbling rates (one bubble per 30 or 60 sec.), this precaution is unnecessary.

The main problem with the maximum bubble-pressure method is knowing whether the gas bubble has the radius of the inside or outside of the capillary or even some intermediate radius. To overcome this difficulty, it is suggested that only capillary materials which are wetted by the liquid should be used so that the inside radius is always operative.

This method is very suitable for the study of aging and other time-dependent phenomena at the gas-liquid interface, but does not lend itself well for studies of viscous melts (e.g. $ZnCl_2$) or molten salts having high vapour pressures. The formation of bubbles may be erratic in such melts.

The experimental apparatus for the maximum bubble-pressure method is simple and easy to operate. The method has a high applicability for high temperature systems. It has also relatively high accuracy at high temperature.

Detachment Methods

A detachment method is any method in which the force required to detach an object from the surface of the liquid is measured. The maximum pull just before the object is detached from the surface, W_{total}, is equated to the weight of the object, W_{object}, plus γL, where L is the total perimeter introduced into the melt.

$$W_{total} = \gamma L + W_{object} \qquad (4)$$

The following four techniques are based on the detachment method:

(a) Wilhelmy Slide Plate Method

The surface tension of a liquid is determined by measuring the force required to detach a plate from the surface of the liquid. The surface tension is calculated by the expression:

$$W_{total} = W_{plate} + 2\ (L + X) \qquad (5)$$

where L is the width of the slide, X is the thickness of the slide, and W_{total}, W_{plate} are observed weights at the moment of detachment and before dipping into liquid respectively.

The following conditions must be satisfied to obtain accurate surface tension values:

(i) the plate must be perfectly wetted so that the contact angle, θ, equals zero;

(ii) the sides of the plate must be vertical;

(iii) the sides of the plate must be flat so that the force acts in a vertical direction.

Similar conditions are also required for the other detachment methods.

(b) Ring Method

The ring method is a variation of the Wilhelmy slide plate method where the plate is replaced by a ring (Fig.3). Surface tension, γ, is calculated by the following equation:

$$M.g = 4\pi R\gamma F \qquad (6)$$

where R is the mean radius of the ring, and F is a dimensionless correction factor introduced by Harkins and Jordan(8). The correction factor F takes into account the small volume of liquid which remains attached to the ring after detachment and the discrepancy between the measured radius R and the true radius of the meniscus R'. F depends only on (R^3/V) and (R/r), where $V = (M/\rho)$ is the volume of liquid held up by the ring and r is the radius of ring wire.

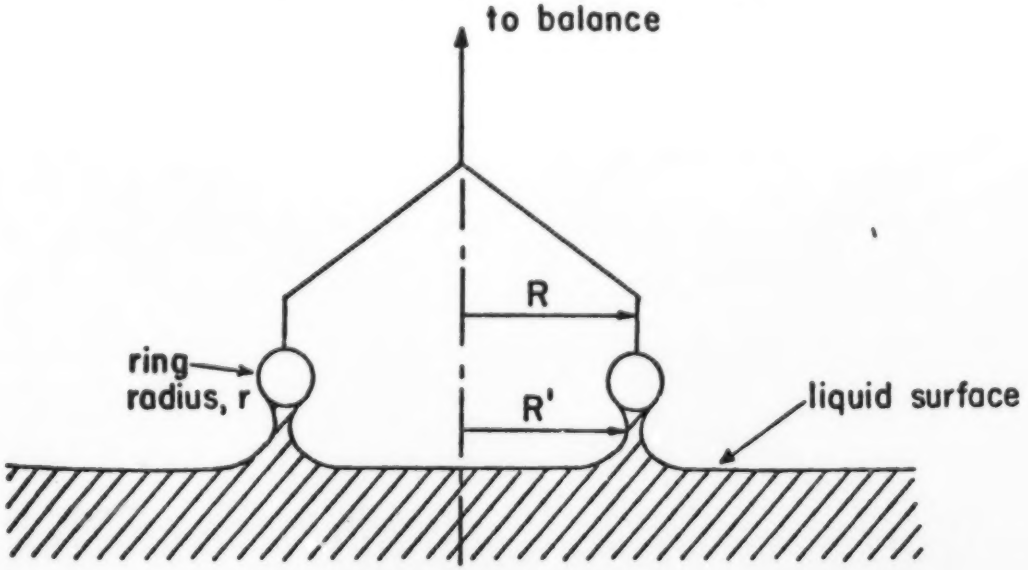

Fig. 3. Ring Method

(c) Cylinder Method

This is a modification of the ring method, the ring being replaced by a vertical hollow cylinder. The surface tension is calculated by a similar equation to that for the ring method:

$$M.g = 4\pi R\gamma F \qquad (7)$$

where R is the mean radius of the cylinder and F is a correction factor depending on (R^3/V) and (R/x). x is the thickness of the cylinder wall.

Liquids of known surface tension are used to obtain the correction factor F(9). The values of F for this method are not the same as for a ring.

The effect of the contact angle, θ, on the maximum pull on the cylinder was investigated by King(9) and found to be negligible unless $\theta > 40^{o}$.

(d) Pin Method

The surface tension detachment pin is part of an Archimedean density bob. The surface tension is calculated by an equation similar to Eq.(7). Densities can also be obtained as part of the same experiment in the conventional manner by complete immersion of the Archimedean bob when the surface tension measurements have been completed.

These detachment methods are based on the assumption of zero contact angle at the moment of detachment. It is advisable that only material which wetted perfectly by the liquid studied is used as a detachment object. For the derivation of the correction factors,

the density values of the liquid are required. When detachment methods are used for high temperature measurements, some experimental difficulties should be taken into account such as condensation of the evaporated liquid on the detachment object and on the wire or rod hanging the object; effects of thermal convective gas flow in the furnace on the reading of balance; change in the bouyant force of gas at higher temperature; and difficulty in locating the exact position of the detachment object. Of the detachment methods, the cylinder technique appears to be most suited for high temperature measurements.

Drop Weight Method

Drops formed extremely slowly at the tip of a capillary detach themselves when the gravitational pull (M.g) just exceeds the counteracting force of the surface tension. Sufficient drops must be collected in a container to accurately determine the average weight per drop.

The mechanism of drop formation does not follow ideal behaviour since part of the liquid remains on the capillary which causes the actual drop weight to be less than the total weight below the capillary. Consequently, Harkins and Brown(10) introduced a correction factor F. Thus, the surface tension is calculated by the following equation:

$$M.g = 2\pi r\gamma F \qquad (8)$$

where M is the weight of drop and r is the capillary radius. They(10) showed the correction factor F was solely dependent on $(r/V)^{1/3}$,

where V (= M/ρ) is the volume of the drop. It requires the knowledge of liquid density ρ to calculate the correction factor.

Drop formation must be controlled to proceed at an extremely slow rate to avoid any errors arising from the downward movement of the liquid. This will be very difficult to do.

Profile Measurement Methods

The general procedure for this class of method is to form a liquid drop or a gas bubble in the liquid studied, and then to make certain measurements of its dimensions.

Since the drop or bubble is not permitted to detach itself, the profile methods are static ones. Thus, errors arising from the movement of the drop or gas are avoided. A major advantage of the profile methods is that they require no correction for the contact angle.

(a) Sessile Drop Method

The drop of liquid is formed on a horizontal flat basal support. Photographs are taken to measure the dimensions of the drop shape. The surface tension of the liquid is calculated using the following equation:

$$\gamma = g.\Delta\rho\ (b^2/\beta) \qquad (9)$$

where $\Delta\rho$ = difference in density between the liquid and gas phase, b = radius of curvature at the apex, and β = shape factor. Both b and β can be obtained from (X/Z) ratio by using Bashforth and Adams' tables(11). (see Fig.4)

It is characteristic for this method that the contact angle, θ, between the liquid and the basal support material can be measured. Once θ is measured, the density of the liquid which is required for the calculation of the surface tension is obtained simultaneously.

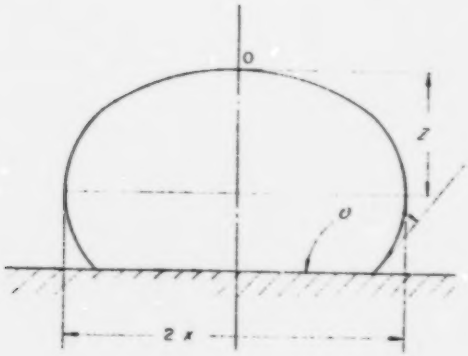

Fig.4. Sessile Drop Method

However, the contact angle, θ, must be larger than 90^{o} to get the desirable drop shape such as shown on Fig.4. When $\theta < 90^{o}$, precise measurement is very difficult. Great care has to also be taken to ensure the symmetry of the drop shape.

The experimental apparatus for the method is relatively simple and easy to operate. It is highly applicable to high temperature systems, especially to the surface tension measurement of molten metals and alloys. Oxide materials are available for the basal support which is not wetted by molten metals and alloys. For molten slags, graphite has been used as a basal support.

(b) Sessile Bubble Method

An alternative arrangement is to form a gas bubble in the liquid and to study its shape in a similar manner (Fig.5). The Bashforth and Adams' tables(11) are used to evaluate the surface tension from the dimensions of bubble.

It is suited for systems where visual observation of the bubble is possible. X-ray apparatus will be needed to photograph the bubble for high temperature systems. The contours of the x-ray photographs are usually too diffuse to obtain accurate measurements. The results are of low precision.

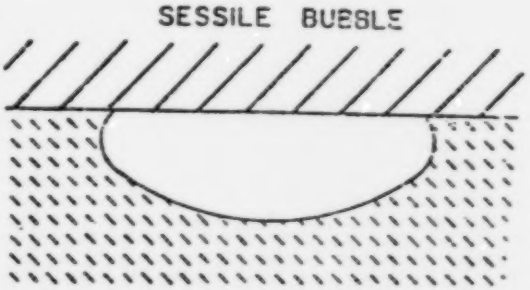

Fig.5. Sessile Bubble Method.

(c) Pendant Drop Method

The procedure with this method is to slowly form a drop of liquid at the tip of a capillary and to measure (from a photograph) the dimensions of the drop profile. A typical pendant drop profile

is shown in Fig.6. The surface tension γ can be calculated by using Eq.(10).

$$\gamma = \Delta\rho.g.de.F \qquad (10)$$

where $\Delta\rho$ is the density difference between the liquid and gas phase and de is the equatorial diameter of the drop. The function F has been obtained and tabulated as a function of (de/ds)(12-16).

The equation demands the knowledge of the liquid density which cannot be obtained by this method. Any inaccuracy in the data must automatically lead to errors in the calculation of the surface tension.

The method allows measurements over a large temperature range, unless the capillary material limits the applicability. This method is also suited to the study of aging (dynamic) effects.

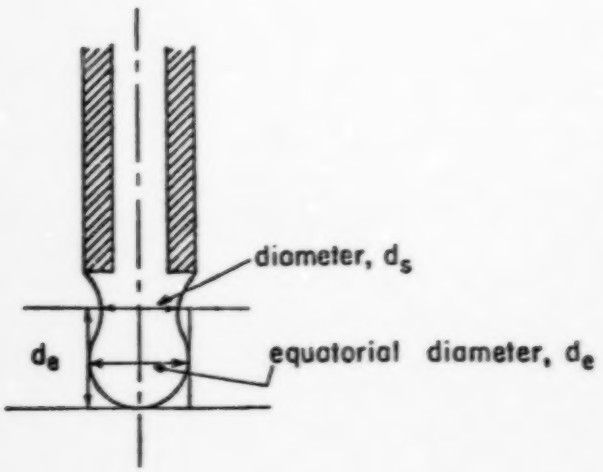

Fig.6. Pendant Drop Method.

CONCLUSIONS

The methods described here require knowledge of the liquid density. Only one exception is the twin capillary method. The maximum bubble-pressure method, pin method, and sessile drop method allow simultaneous density measurements.

The methods which are applicable for high temperature measurements are the maximum bubble-pressure method, sessile drop method, cylinder method, pendant drop method, and drop weight method. According to Boni and Derge(17), the order of the high temperature applicability is as follows:

(1) maximum bubble-pressure method; (2) cylinder method;
(3) sessile drop method.

Most of the recent surface tension studies of molten salts and slags have been done by using the maximum bubble-pressure method. This method showed high accuracy and reproducibility for high temperature measurements. The experimental apparatus for the method is relatively simple and easy to operate. The principles of maximum bubble-pressure method have been well established theoretically. It is concluded that the most versatile method and one that is applicable to the molten salt systems at high temperature is the maximum bubble-pressure method.

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A REVIEW OF THE SURFACE TENSION MEASUREMENTS OF THE TERNARY SYSTEMS KCl-NaCl-$PbCl_2$ AND KCl-LiCl-$PbCl_2$

In general, very little data of any kind exist on the surface tension of ternary salt systems. None was found for the two systems KCl-NaCl-$PbCl_2$ and KCl-LiCl-$PbCl_2$. However, the surface tension data of the pure salts and of the binaries making up the respective ternaries appear to be well established. These data have been critically reviewed by Janz et al(1,2). The recommended values of the surface tension of interest are summarized below.

PURE SALTS

(a) KCl

The surface tension of pure KCl has been studied by a number of investigators(3-15). Table I shows a summary of the temperature range, capillary and cell material used by these investigators. Janz et al(2) have critically evaluated these results and arrived at an average equation for the surface tension of KCl, as given by Eq. (1).

$$\gamma_{KCl} = 1.79.1168 - 0.76026 \times 10^{-1} T \ (\text{dyn cm}^{-1}) \qquad (1)$$

This equation is based on the studies of Ellis and Freeman(3), Matsumura et al(4), Grjotheim et al(5), Lehman(13), Smirnov and Stepanov(15), Bloom et al(16), Jaeger(17) and Peake and Bothwell(11). All these investigators used the maximum bubble pressure method except Grjotheim et al(5), who used the pin detachment method.

TABLE I

SURFACE TENSION STUDIES OF KCl

Temperature Range T	Cell Material	Reference
1043-1273	Pt	3
1073-1173	Pt	4
1073-1273	Pt	5
1073	Pt	6
800-950	Pt	7
800-850	Pt	8
850	Pt	9
800-900	Pt	10
830-920	90% Pt - 10% Rh alloy capillary and porcelain crucible	11
730-930	90% Pt - 10% Rh alloy capillary and graphite crucible	12
828-918	90% Pt - 10% Rh alloy capillary and alumina crucible	13
800-900	Pt	14
1073-1193	Pt	15

The quoted precision and uncertainty of Eq. (1) are ±0.26% and ±0.5% respectively.

(b) LiCl

The recommended equation for the surface tension of LiCl is

$$\gamma_{LiCl} = 180.42 - 5.83 \times 10^{-2} T \ (dyn\ cm^{-1}) \qquad (2)$$

(precision ±0.3%, uncertainty ±0.5%)

This equation is based on the work of Ellis(18). The technique adopted by this investigator was the maximum bubble pressure method.

(c) NaCl

The surface tension of NaCl is well established and the recommended equation is based on the maximum bubble pressure method used by Sokolova and Voskresenskaya(19).

$$\gamma_{NaCl} = 191.4 - 7.19 \times 10^{-2} T \ (dyn\ cm^{-1}) \qquad (3)$$

The precision and uncertainty associated with this equation are ±0.2% and ±1% respectively.

(d) $PbCl_2$

Of the three investigations reported on the surface tension of $PbCl_2$(20,21,22), the recommended value is based on the work of Dahl and Duke(22). The main problem encountered with surface tension measurements of $PbCl_2$ is one of volatility. The recommended equation is

$$\gamma_{PbCl_2} = 233.6 - 12.4 \times 10^{-2} T \ (dyn\ cm^{-1})$$

The maximum bubble pressure method was also used in the recommended value.

BINARY SYSTEMS

(a) KCl-LiCl

The KCl-LiCl has been studied by three investigators(23,24,4) all using the maximum bubble pressure technique. Of these, Janz et al(2) recommend the work of Nissen and Carlsten(23,24).

The temperature dependent equations at experimental compositions are summarized in Table II.

(b) KCl-NaCl

Seven studies are reported(4,6,7,10,25,26,27). All these investigators used the maximum bubble pressure technique. Of these, Janz et al(2) have selected the work of Desyatnikov(7), who used platinum capillary and a quartz crucible. The recommended temperature dependent equations are given in Table III.

(c) $KCl-PbCl_2$

Of the three reported studies(10,22,28), Janz et al(2) recommend the data of Dahl and Duke(10). The technique adopted by these authors was the maximum bubble method using a Pt-10% Rh capillary in a platinum crucible. The temperature dependent equations are given in Table IV.

TABLE II

TEMPERATURE DEPENDENT EQUATIONS

$\gamma = a + b\,T$ (dyn cm^{-1})

for the KCl-LiCl System

Mol % LiCl	a	b x 10^2
10	187.4541	-8.30993
15	189.5651	-8.49146
22	196.2269	-9.16605
30	190.9364	-8.69217
40	189.4704	-8.45251
50	187.3435	-8.08096
58	189.5657	-8.23478
69	188.3650	-7.82852
79	183.5300	-7.10111
90	183.3852	-6.75324

TABLE III

TEMPERATURE DEPENDENT EQUATIONS

$\gamma = a + b\,T$ (dyn cm^{-1})

for the KCl-NaCl System

Mole % NaCl	a	b x 10^2
0	176.8	-7.5
10	178.1	-7.5
25	178.0	-7.4
40	177.3	-7.2
50	179.3	-7.2
60	180.4	-7.2
75	181.2	-7.0
90	183.5	-6.8
100	187.0	-6.8

TABLE IV

TEMPERATURE DEPENDENT EQUATIONS

$\gamma = a + b\,T$ (dyn cm^{-1})

for the $KCl-PbCl_2$ System

Mole % $PbCl_2$	a	b x 10^2
31.77	169.9381	- 7.3489
36.86	180.9981	- 8.5499
48.85	196.6602	-10.5613
49.13	193.1133	-10.0855
64.12	193.7382	- 9.8527
75.68	201.4260	-10.4167
100.00	233.6772	-12.4284

(d) $LiCl-PbCl_2$

Two studies exist for the $LiCl-PbCl_2$ system(10,22). The recommended data is based on the maximum bubble pressure method adopted by Dahl and Duke(10) using a Pt-10% Rh capillary in a melt contained in a platinum crucible. The temperature dependent equations are given in Table V.

TABLE V

Temperature Dependent Equations

$\gamma = a + b\,T$ (dyn cm^{-1})

for the $LiCl-PbCl_2$ System

Mole % $PbCl_2$	a	b x 10^2
26.37	200.9088	- 8.8542
31.26	211.7828	-10.2165
49.63	194.0891	- 7.9463
50.61	209.6978	- 9.8480
64.25	226.3552	-11.8665
69.90	200.8931	- 8.7107
100.00	233.6772	-12.4284

(e) $NaCl-PbCl_2$

There are four surface tension studies reported for this system(6,10,20,22). Of these, Janz et al recommend the data of Bloom et al(20) who also used the maximum bubble pressure technique.

The cell materials were a Pt - 10% Rh capillary. The recommended temperature-dependent equations are shown in Table VI.

TABLE VI

TEMPERATURE DEPENDENT EQUATIONS

$\gamma = a + b\,T$ (dyn cm^{-1})

for the $NaCl-PbCl_2$ System

Mole % $PbCl_2$	a	b . 10^2
0	216.2	- 9.3
24.8	212.6	-10.0
50.0	214.5	-10.7
75.0	207.5	-10.2
100.0	222.2	-11.0

ESTIMATION OF SURFACE TENSION IN THE TERNARY SYSTEMS, $PbCl_2$-KCl-NaCl AND $PbCl_2$-KCl-LiCl

In a previous study concerned with the density measurements in the $PbCl_2$-KCl-NaCl and $PbCl_2$-KCl-LiCl systems(29), it was found that a general additivity relation of the binary systems was applicable to a first approximation. Based on the knowledge, an estimation is proposed of the surface tensions within the same ternary systems.

This estimation is based on the addivity equation

$$\gamma_{123(y)} = (1-t)\ \gamma_{13(y)} + t\ \gamma_{12(y)}$$

where 1 = $PbCl_2$

2 = KCl

3 = NaCl or LiCl

$$y = 1 - X_{PbCl_2}$$

$$t = \frac{X_{MCl}}{X_{MCl}+X_{KCl}}$$

where M = Li or Na

In these equations, X_i is the mole fraction of component i.

The estimated surface tensions are listed in Tables VII and VIII.

TABLE VII

$PbCl_2$-KCl-NaCl

Estimated Surface Tensions (dyn cm^{-1})

	y					
t	1	0.8	0.6	0.4	0.2	0
0	120.3	114.0	114.0	116.5	122.5	137.7
0.25	123.5	119.3	118.7	120.0	124.5	137.7
0.50	124.8	124.5	123.5	123.5	126.4	137.7
0.75	129.0	129.8	128.2	127.0	128.4	137.7
1	135.8	135.1	133.0	130.5	130.4	137.7

TABLE VIII

$PbCl_2$-KCl-LiCl

Estimated Surface Tensions

(dyn cm^{-1})

t	y = 1	0.8	0.6	0.4	0.2	0
0	120.3	114.0	114.0	116.5	122.5	137.7
0.25	123.5	118.7	118.8	120.8	125.5	137.7
0.50	125.0	123.5	123.6	124.9	128.5	137.7
0.75	129.0	128.2	128.4	129.1	131.5	137.7
1	135.4	133.0	133.2	133.3	134.5	137.7

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